FOAM REPLICATED POROUS 316L STAINLESS STEEL BASED ON TAGUCHI METHOD FOR BIOMEDICAL APPLICATIONS

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"My dearest mum, family, and friends"

This is for all of you

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ABSTRACT

The mismatch between elastic modulus of metal implants and bones which is also known as stress shielding, remains an unresolved issue. Porous metals are one of the most effective ways of reducing stiffness mismatches and achieving stable long-term fixation via full bone in-growth. In this work, porous SS316L is produced using the foam replication technique. The samples were each produced with different compositions of SS316L powders and sintered at various sintering parameters including the sintering temperature, sintering time, heating rate and cooling rate. Scanning electron microscopy (SEM) was used to characterise the microstructure while a compression test was used to determine the mechanical properties of the samples. The physical properties including porosity and density were measured according to the Archimedes principles. The biocompatibility test showed that the porous SS316L produced, exhibited no cytotoxicity reactivity. Furthermore, the optimisations of the sintering parameters were performed using the Taguchi method. The optimised porosity of porous SS316L prepared by ball milling method was 85.44% and achieved using sintering time of 60 minutes, sintering temperature of 1200°C, heating rate of 1°C/min, SS316L composition of 60 wt% and cooling rate of 1°C/min. Whereas, for samples prepared by mechanical stirring method, the optimum porosity was 79.46% and occurred for the samples sintered within 60 minutes at 1200°C of sintering temperature, with the cooling and heating rates of 1°C/min and 2°C/min respectively, and prepared with 70 wt% of SS316L composition. In addition, porous SS316L prepared by ball milling method with modulus of elasticity of 0.08 GPa was obtained by using optimum sintering temperature of 1250°C, sintering time of 60 minutes, heating rate of 2°C/min, SS316L composition of 65 wt% and cooling rate of 1°C/min. Whereas, the modulus of elasticity of 0.05 GPa for porous SS316L prepared by mechanical stirring method was obtained by using the optimum cooling rate of 5°C/min, sintering temperature of 1200°C, sintering time of 120 minutes, SS316L composition of 70 wt% and heating rate of 0.5°C/min respectively. Following optimisation, the porous SS316L produced was found to have attractive mechanical and physical properties much like human bone. Notwithstanding, this included interconnected and open porosity in the range of 79.46 to 85.44 %, density in the range of 1.53-1.76 g/cm^3, pore size in the range of 247–470 µm, modulus of elasticity in the range of 0.05-0.08 GPa, yield strength in the range of 0.52–0.82 MPa and compression strength in the range of 35.87-64.43 MPa.

ABSTRAK

Perbezaan modulus elastik antara bahan implan dan tulang yang juga dikenali sebagai kesan perisai tegasan merupakan masalah yang masih belum selesai. Logam berbusa adalah salah satu pendekatan yang berkesan untuk menangani masalah ini supaya ia sesuai untuk aplikasi jangka panjang melalui pertumbuhan tulang sepenuhnya. Dalam kajian ini, SS316L berbusa telah dihasilkan dengan menggunakan teknik replikasi Poliurethena (PU) berbusa. Sampel dihasilkan dengan komposisi serbuk SS316L yang berbeza, dan disinter pada pelbagai parameter persinteran termasuk suhu persinteran, masa persinteran, kadar pemanasan dan kadar penyejukan. Pengimbasan mikroskop elektron (SEM) digunakan untuk mengkaji mikrostruktur manakala ujian mampatan dijalankan untuk menentukan sifat-sifat mekanik sampel. Ciri-ciri fizikal iaitu keliangan dan ketumpatan diukur menggunakan prinsip Archimedes. Ujian biokompatibiliti telah menunjukkan bahawa SS316L berbusa yang dihasilkan tidak menunjukkan reaktiviti sitotoksik. Selain itu, pengoptimuman parameter persinteran dilakukan dengan menggunakan kaedah Taguchi. Keliangan optimum untuk SS316L berbusa yang disediakan dengan kaedah pengisaran bebola adalah 85.44% dan diperoleh dengan menggunakan masa persinteran selama 60 minit pada suhu 1200°C dengan kadar pemanasan 1°C/min, disediakan dengan komposisi SS316L sebanyak 60 wt% serta disejukkan dengan kadar penyejukan pada 1°C/min. Manakala untuk sampel yang disediakan dengan kaedah pengadukan mekanikal, keliangan optimum adalah 79.46% dan dicapai setelah disinter selama 60 minit pada suhu 1200°C, dengan kadar penyejukan dan pemanasan masing-masing pada 1°C/min dan 2°C/min, dan disediakan dengan komposisi SS316L sebanyak 70 wt%. Di samping itu, SS316L berbusa yang disediakan dengan kaedah pengisaran bebola dengan modulus keanjalan optimum, 0.08 GPa diperoleh dengan menggunakan suhu persinteran pada 1250°C, masa persinteran selama 60 minit, kadar pemanasan pada 2°C/min, komposisi SS316L sebanyak 65 wt% dan dengan kadar penyejukan pada 1°C/min. Manakala modulus keanjalan optimum, 0.05 GPa untuk SS316L berbusa yang disediakan oleh kaedah pengadukan mekanikal pula diperoleh dengan menggunakan kadar penyejukan optimum pada 5°C/min, suhu persinteran pada 1200°C, masa persinteran selama 120 minit, komposisi SS316L sebanyak 70 wt% dan kadar pemanasan pada 0.5°C/min. Selepas pengoptimuman, SS316L berbusa yang dihasilkan didapati mempunyai sifat mekanikal dan fizikal yang hampir sama dengan tulang manusia. Walau bagaimanapun ini termasuk keliangan terbuka yang saling berkait dalam lingkungan 79.46-85.44%, ketumpatan di antara 1.53 -1.76 g/cm^3, saiz liang di antara 247-470 μm, modulus keanjalan di antara 0.05-0.08 GPa, kekuatan alah di antara 0.52-0.82 MPa dan kekuatan mampatan dalam lingkungan 35.87-64.43 MPa.

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LIST OF ABBREVIATION

ASTM	-	American Society for Testing and Materials
CMC	-	carboxymethyl cellulose
Cps	-	Centipoise
DF	-	Degrees of freedom
DIN	-	German Institute for Standardisation
DOE	-	Design of experiments
ECHC	-	European Commission on Health and Consumers
EDX	-	Energy Dispersive X-Ray
FDA	-	U.S Food and Drug Administration
GPa	-	Gigapascal
ISO	-	International Organization for Standardization
MPa	-	Megapascal
MPIF	-	Metal Powder Industries Federation
SEM	-	Scanning electron microscope
SS316L	-	Stainless steel 316L
PEG	-	Polyethylene glycol
PM	-	Powder Metallurgy
PU	-	Polyurethane
PVA	-	Polyvinyl Alcohol
SBF	-	Simulated Body Fluid
S/N	-	Signal to Noise Ratio
TGA	-	Thermal gravimetric analysis
UTS	-	Ultimate tensile stress
WHO	-	World Health Organization
wt%	-	Weight percentage
SLM	-	Selective Laser Method

LIST OF SYMBOLS

a	-	samples width
b	-	samples length
c	-	samples height
A1	-	1°C/min (Cooling Rate)
A2	-	5°C/min (Cooling Rate)
B1	-	1200°C (Sintering Temperature)
B2	-	1250°C (Sintering Temperature)
B3	-	1300°C (Sintering Temperature)
C1	-	0.5 °C/min (Heating Rate)
C2	-	1 °C/min (Heating Rate)
C3	-	2 °C/min (Heating Rate)
D1	-	60 minutes (Sintering Time)
D2	-	90 minutes (Sintering Time)
D3	-	120 minutes (Sintering Time)
E1	-	60 wt. % SS316L (SS316L composition)
E2	-	65 wt. % SS316L (SS316L composition)
E3	-	70 wt.% SS316L (SS316L composition)
E	-	Modulus of elasticity
σ_c	-	Compression Strength
σ_y	-	Yield Strength
σ	-	Stress
ε	-	Strain
ρ	-	Density
H_2O	-	Water
V	-	Volume

W_{dry}	-	dry weight
W_{sub}	-	submerged weight
W_{wet}	-	wet weight

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The mechanical failure of the human body can often be repaired by the surgical implantation of synthetic replacement parts called biological implants. Numerous researches have been conducted over time by the clinicians and engineers to study the physical and mechanical properties of all types of human bones and implants to treat various injuries involving bone replacement. The success of bone replacement with implant depends on many factors such as physical and mechanical properties of the implant material, biocompatibility between a human body with the implant material, patient's health condition and expertise of the surgeon who performs the surgery. At present, the implant material is only able to survive and work well within the human body for about 12 to 15 years. This condition causes re-surgery is needed to monitor the condition of the implant, the patient's health and to replace the implant. Re-surgery and replacement of the implants will involve additional cost to the patient. The cause of the failure of an implant is varies, including the mechanical, chemical, biocompatibility, implant design, surgery, tribology and so forth [1].

Since 1940s, experts from various fields including science, engineering, and medicine have introduced several new processing methods, design concepts, and surgical techniques. Although the 20th century saw many inventions and advances in the development of biomaterials with its own characteristics, however, to date, there is still no implant material that exactly matches the composition, structure, and property of any part of the human body. In addition, in recent years, nanotechnology, biomimetic, and tissue engineering concepts have rapidly developed and became a new boundary in the development of nanoscale biomaterials [2].

The use of metals as biomedical implants primarily stainless steel, Co-based alloys, Ti and its alloys to replace damaged or failed tissue has begun since the early 1900s. Stainless steel is the first metal implant used successfully in the field of surgery. However, the elastic modulus of stainless steel and Co-Cr alloys is higher than that of the natural bone which is about ten times larger, resulting in some complications of mechanical instability and the structure between the implants and hose tissues. The elastic modulus of Ti and its alloy is found to be about five times larger than the natural bone. If a stiffer implant is inserted into hard tissues (eg, Bones), the bones will undergo a reduced mechanical stress which gradually leads to bone absorption. Therefore, this phenomenon is known as a "stress shielding effect" that leads to the death of bone cells [3]. Therefore, low stiffness cellular metals have been produced to overcome this problem.

In the 1970s, the use of porous materials with open pore structure as bone implant has been introduced. Accordingly, this open pore structure promotes the integration of both bones and blood vessels and overcomes the large elastic modulus difference between the bones and the implants. Additionally, porous metals are able to reach similar strength with cancellous bone because of this porous structure. Thus, porous metals have attracted significant attention among medical researchers all over the world due to these unique properties [4].

In order to replace the cancellous bone, there are basically some features that need to be fulfilled by the metal implants which include of having interconnected pores about 30-90% with a pore size in the range of 100-600 μm to provide space for cell migration and new tissue in-growth, and a low Young modulus that similar to the cancellous bone, <3 GPa [5].

1.2 Problem Statement

There are two types of orthopedic implants which are temporary implant and permanent implant. Plates and screws are examples of the temporary implant. While for a permanent implant, it usually involved with knees, shoulders, spine, hips, fingers and feet replacements. In the case of a permanent implant, it is very important to ensure that the bonding between the implant material and the living tissue is strong enough and safe. This strong bonding can be achieved through the tissue in-growth within the open and interconnected pores of the implant materials [6]. However, the current metal implant still has some weaknesses. First, the bonding strength is still not high enough and needs to be improved. Second, the large difference of elastic modulus between the bone and implant should be reduced.

Figure 1.1 shows the elastic modulus of most materials that currently used for biomedical applications. From this diagram, it is clearly shows that the elastic modulus of cancellous and cortical bone is very low compared to the elastic modulus of metal implants, especially in the case of stainless steel. This will cause stress shielding to occur on the interface that will affect the long-term stability of the implant [7]. This stress shielding problem still remains as an issue of attention among researchers around the world. In fact, the use of porous materials as implant materials also attracts researchers' interest and attention as a very effective method to reduce excessive stiffness and modulus of elasticity to achieve long-term stability. The elastic modulus of porous metal implants can be modified to match the human bone and thus help prevent the effect of stress shielding on the bones and implants. In addition, porous metal can provide space for bone ingrowth to achieve biological fixation.

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